# Quality Control of Lubricants

Fast and chemical-free determination of the Acid Number, viscosity, moisture content, and color number of lubricants

For lubricant analysis, determination of the Acid Number (ASTM D664), viscosity (ASTM D445), moisture content (ASTM D6304), and color number (ASTM D1500) require the use of multiple analytical technologies and, in part, large volumes of chemicals. Time to result can therefore be quite a long and costly process.

This application note demonstrates that the XDS RapidLiquid Analyzer operating in the visible and near-infrared spectral region (Vis-NIR) provides a fast and cost-efficient alternative for the determination of the AN, viscosity, moisture content, and color number of lubricants. With **no sample preparation or chemicals needed**, Vis-NIR spectroscopy allows for multi parameter analysis of lubricants in **less than one minute**.

Ω Metrohm

## EXPERIMENTAL EQUIPMENT

Lubricant samples were measured with a XDS RapidLiquid Analyzer in transmission mode over the full wavelength range (400–2500 nm) (**Figure 1**). Reproducible spectrum acquisition was achieved using the instrument's built-in temperature control (at 40°C). For convenience, disposable vials with a path length of 8 mm were used, which made cleaning of the sample vessels unnecessary. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.



**Figure 1.** XDS RapidLiquid Analyzer and lubricant samples.

*Table 1.* Hardware and software equipment overview

| Equipment | Metrohm number |
|---|---|
| XDS RapidLiquid Analyzer | 2.921.1410 |
| Disposable vials, 8 mm diameter, transmission | 6.7402.000 |
| Vision Air Complete | 6.6072.208 |

## RESULT

The obtained Vis-NIR spectra (**Figure 2**) were used to create prediction models for quantification of the Acid Number, viscosity, moisture content, and color number in lubricants. Correlation diagrams, which display the relationship between Vis-NIR prediction and primary method values, are used to determine the quality of the prediction models. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.
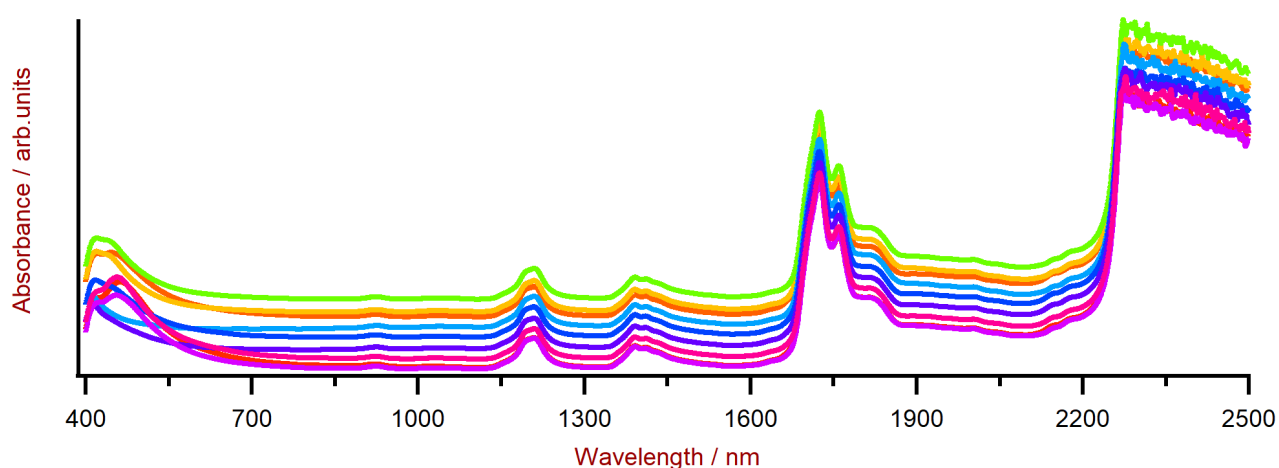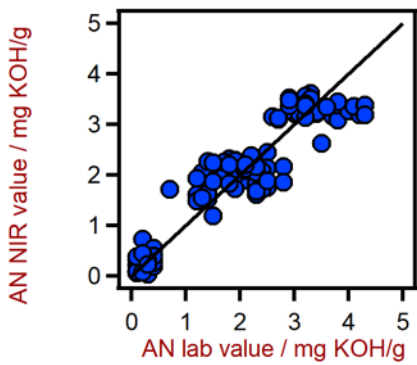


**Figure 2.** Selection of lubricant oil Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm disposable vials. For display reasons a spectra offset was applied.
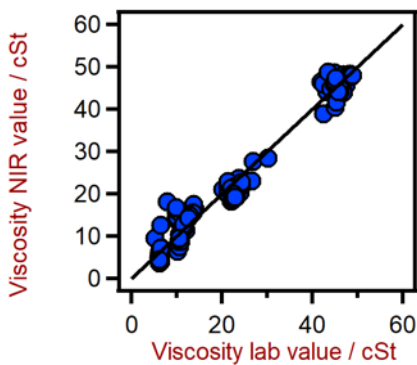
## RESULT ACID NUMBER



| Figures of Merit | Value |
|---|---|
| $R^2$ | 0.898 |
| Standard Error of Calibration | 0.422 mg KOH/g |
| Standard Error of Cross-Validation | 0.439 mg KOH/g |

**Figure 3.** Correlation diagram and the respective figures of merit for the prediction of the Acid Number (AN) in lubricants using a XDS RapidLiquid Analyzer. The AN lab value was evaluated using titration.
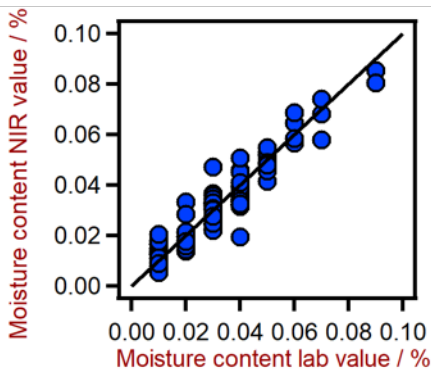
## RESULT VISCOSITY



| Figures of Merit | Value |
|---|---|
| $R^2$ | 0.700 |
| Standard Error of Calibration | 0.841 |
| Standard Error of Cross-Validation | 0.916 |

**Figure 4.** Correlation diagram and the respective figures of merit for the prediction of the viscosity in lubricants using a XDS RapidLiquid Analyzer. The viscosity lab value was evaluated using viscometry.
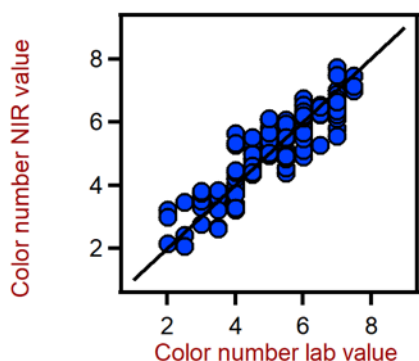
## RESULT MOISTURE CONTENT



| Figures of Merit | Value |
|---|---|
| $R^2$ | 0.907 |
| Standard Error of Calibration | 0.0059 % |
| Standard Error of Cross-Validation | 0.0062 % |

**Figure 5.** Correlation diagram and the respective figures of merit for the prediction of the moisture content in lubricants using a XDS RapidLiquid Analyzer. The moisture content lab value was evaluated using Karl Fischer (KF) titration.

Ω Metrohm

## RESULT COLOR NUMBER



| Figures of Merit | Value |
|---|---|
| $R^2$ | 0.700 |
| Standard Error of Calibration | 0.841 |
| Standard Error of Cross-Validation | 0.916 |

**Figure 6.** Correlation diagram and the respective figures of merit for the prediction of the color number in lubricants using a XDS RapidLiquid Analyzer. The hydroxyl number lab value was evaluated using photometry.

## CONCLUSION

The following application note demonstrates the feasibility of NIR spectroscopy for the analysis of key quality parameters in lubricants. In comparison to wet chemical methods (**Table 2**), the time to result is a major advantage of NIR spectroscopy, since all parameters are determined **in a single measurement in less than a minute**.

*Table 2.* Time to result overview for the different quality control parameters.

| Parameter | Method | Time to result |
|---|---|---|
| Acid Number | Titration | ~5 min |
| Viscosity | Viscometry | ~4 min |
| Moisture content | KF Titration | ~5 min |
| Color number | UV-Vis Photometer | ~1 min |

**Analytes:** Acid Number, viscosity, moisture content, color number
**Matrix:** Lubricants
**Industry:** Petrochemical
**Standards:** ASTM E1655

Ω Metrohm